

Advanced Polymer Chromatography - Method Development Tools for SEC Analysis of PEG

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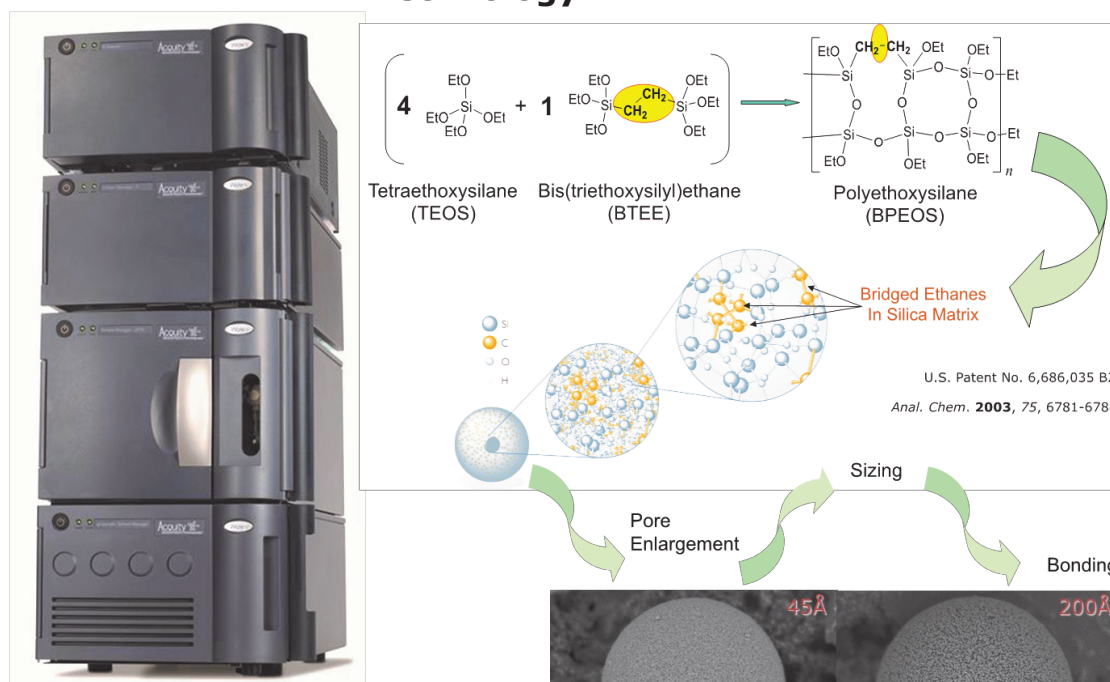
INTRODUCTION

Traditionally, size exclusion or gel permeation chromatography (SEC/GPC) is used for the characterization of polymeric material, specifically their molecular weight distribution. In order to resolve polymeric species, long column lengths and banked column configurations are commonly used, resulting in lengthy analytical test cycle times as well as the associated consumption of costly and often hazardous solvents. Additionally, many test sets suffer from minimal replicate data points due to the typical analysis time resulting data with limited statistical weighting.

In this paper the benefits of a comprehensive systematic approach for polymer molecular weight characterization will be presented. Waters® ACQUITY® Advanced Polymer Chromatography® (APC™) System, with its innovative and robust ACQUITY APC™ column technology, allows for improved resolution of polymer distributions with significantly shorter chromatographic total analysis cycle times. Additionally, we will describe some of the tools that are available to develop stable and impactful test methods that result in richer data sets based on more stable operating conditions, and replicate analyses that are easily obtained within minutes and not hours.

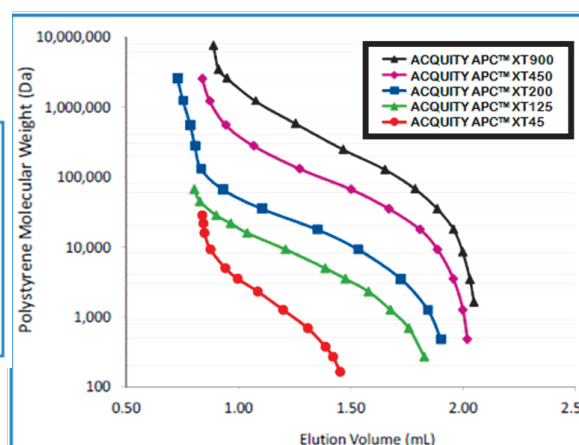
METHODS

Advanced Polymer chromatography system and BEH particle Technology



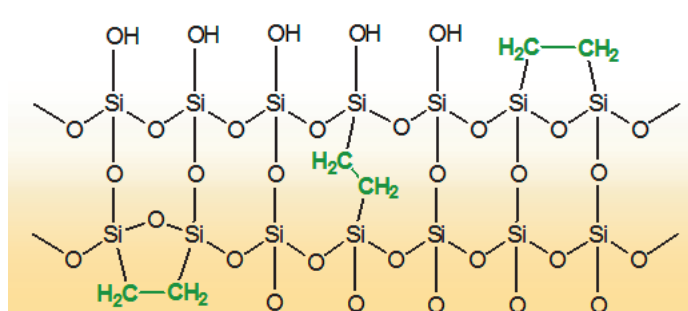
- Five pore sizes
 - 45 Å (200 - 5,000) 1.7µm
 - 125 Å (1,000 - 30,000) 2.5µm
 - 200 Å (3,000 - 70,000) 2.5µm
 - 450 Å (20,000 - 400,000) 2.5µm
 - 900 Å (80,000 - 2,000,000) 2.5µm

- Three column lengths
 - 30 mm
 - 75 mm
 - 150 mm

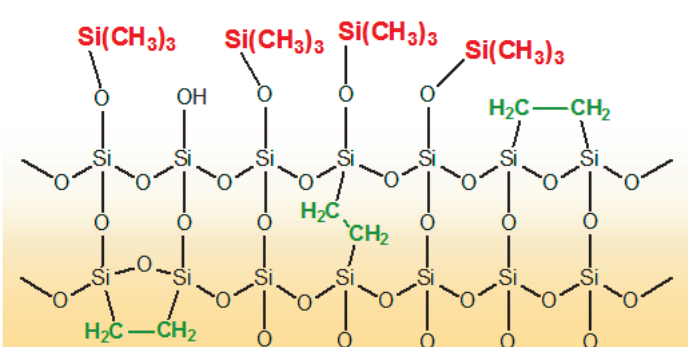


Surface Chemistry

AQ columns are unbonded

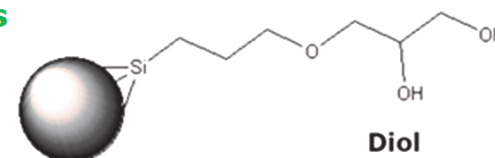


XT columns are TMS bonded



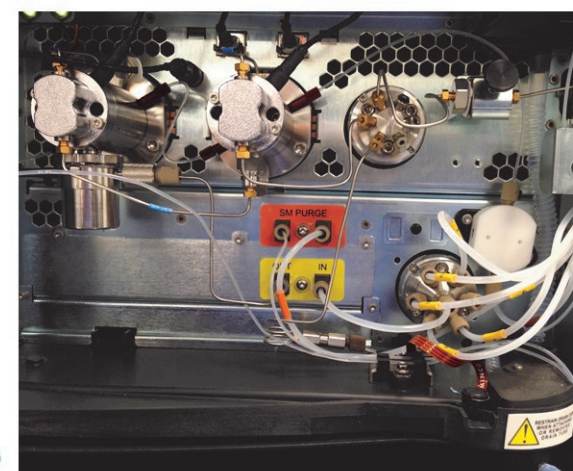
BEH Protein SEC Columns

125A 1,000 - 80,000
200A 10,000 - 450,000
450A 100,000 - 1,500,000



Automating the Method Screening Process

- Optional solvent select valve
- Fully compatible with the ACQUITY APC supported solvents
 - Aqueous and organic
 - 6 solvent lines
- Enables automated solvent switching on the ACQUITY APC System
 - Solvent resilient ACQUITY APC hybrid particles not susceptible to swelling with solvent switching

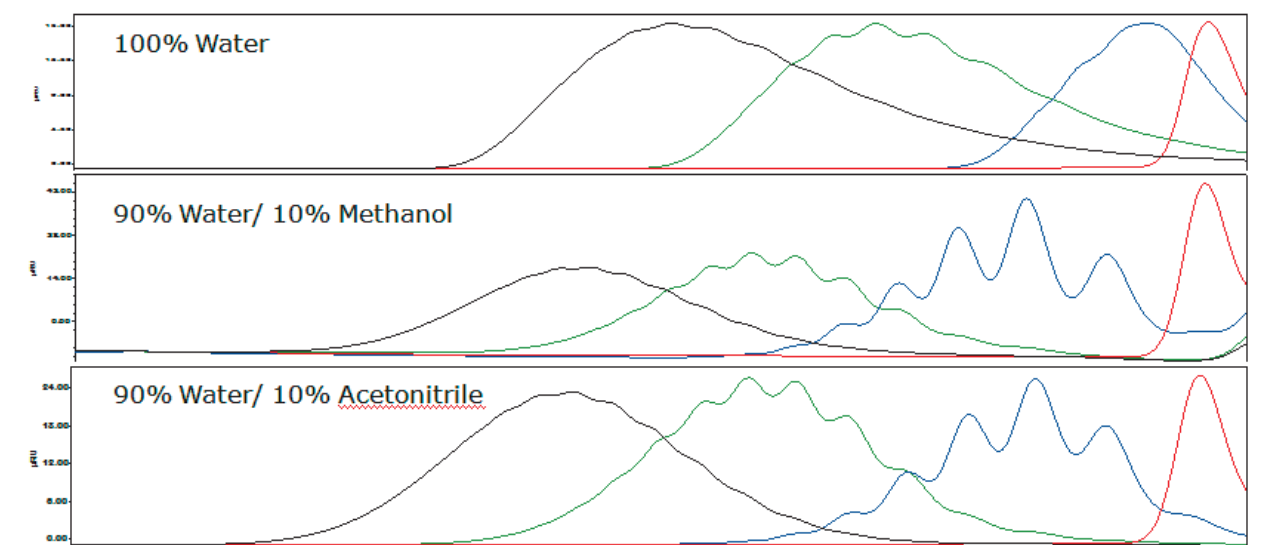


APC Method Development Screening Protocol— Completed in 4 hours

| E | Plate/Well | Inj Vol (µL) | # of Injs | Sample Name | Function | Run Time (Minutes) | Method Set / Report Method | Processing |
|----|------------|--------------|-----------|----------------------|-------------------------|--------------------|----------------------------|------------|
| 1 | | | | | Condition Column | 45.00 | APC AQ 40C S1 ACN 750 | |
| 2 | | | | | Purge Det | 5.00 | APC AQ 40C S1 ACN 750 | |
| 3 | | | | | Equilibrate | 10.00 | APC AQ 40C S1 ACN 750 | |
| 4 | 1A,1 | 5.0 | 1 | Blank | Inject Samples | 5.00 | APC AQ 40C S1 ACN 750 | Normal |
| 5 | 1B,1 | 5.0 | 1 | PEG 106 10% ACN 40C | Inject Narrow Standards | 5.00 | APC AQ 40C S1 ACN 750 | Normal |
| 6 | 1B,2 | 5.0 | 1 | PEG 202 10% ACN 40C | Inject Narrow Standards | 5.00 | APC AQ 40C S1 ACN 750 | Normal |
| 7 | 1B,3 | 5.0 | 1 | PEG 430 10% ACN 40C | Inject Narrow Standards | 5.00 | APC AQ 40C S1 ACN 750 | Normal |
| 8 | 1B,4 | 5.0 | 1 | PEG 633 10% ACN 40C | Inject Narrow Standards | 5.00 | APC AQ 40C S1 ACN 750 | Normal |
| 9 | | | | | Equilibrate | 45.00 | APC AQ 40C S2 MeOH 750 | |
| 10 | | | | | Purge Det | 5.00 | APC AQ 40C S2 MeOH 750 | |
| 11 | | | | | Equilibrate | 10.00 | APC AQ 40C S2 MeOH 750 | |
| 12 | 1C,1 | 5.0 | 1 | PEG 106 10% MeOH 40C | Inject Narrow Standards | 5.00 | APC AQ 40C S2 MeOH 750 | Normal |
| 13 | 1C,2 | 5.0 | 1 | PEG 202 10% MeOH 40C | Inject Narrow Standards | 5.00 | APC AQ 40C S2 MeOH 750 | Normal |
| 14 | 1C,3 | 5.0 | 1 | PEG 430 10% MeOH 40C | Inject Narrow Standards | 5.00 | APC AQ 40C S2 MeOH 750 | Normal |
| 15 | 1C,4 | 5.0 | 1 | PEG 633 10% MeOH 40C | Inject Broad Samples | 5.00 | APC AQ 40C S2 MeOH 750 | Normal |
| 16 | | | | | Equilibrate | 45.00 | APC AQ 40C S3 H2O 750 | |
| 17 | | | | | Purge Det | 5.00 | APC AQ 40C S3 H2O 750 | |
| 18 | | | | | Equilibrate | 10.00 | APC AQ 40C S3 H2O 750 | |
| 19 | 1D,1 | 5.0 | 1 | PEG 106 100% H2O 40C | Inject Narrow Standards | 5.00 | APC AQ 40C S3 H2O 750 | Normal |
| 20 | 1D,2 | 5.0 | 1 | PEG 202 100% H2O 40C | Inject Narrow Standards | 5.00 | APC AQ 40C S3 H2O 750 | Normal |
| 21 | 1D,3 | 5.0 | 1 | PEG 430 100% H2O 40C | Inject Narrow Standards | 5.00 | APC AQ 40C S3 H2O 750 | Normal |
| 22 | 1D,4 | 5.0 | 1 | PEG 633 100% H2O 40C | Inject Broad Samples | 5.00 | APC AQ 40C S3 H2O 750 | Normal |
| 23 | 1A,1 | 5.0 | 1 | Blank | Inject Samples | 5.00 | APC AQ 40C S3 H2O sd | Normal |

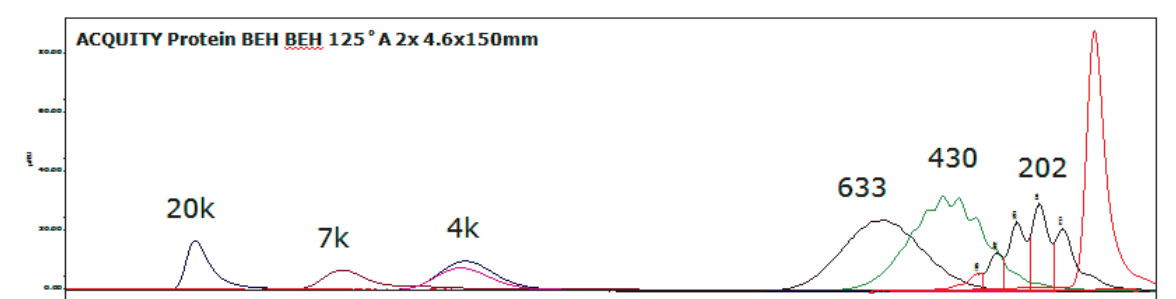
RESULTS

PEG Method Screening Results

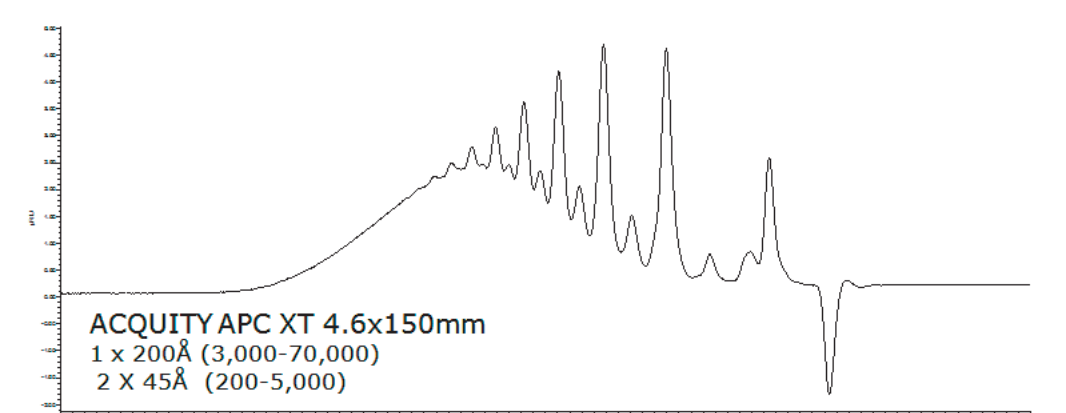


Column: Two 4.6x150mm 1.7µm 125A° BEH Protein SEC; Column Temp: 40°C
Flow Rate: 0.75mL/min; Standards: PEG 106, 202, 430, 633; System backpressure ~ 12,000psi

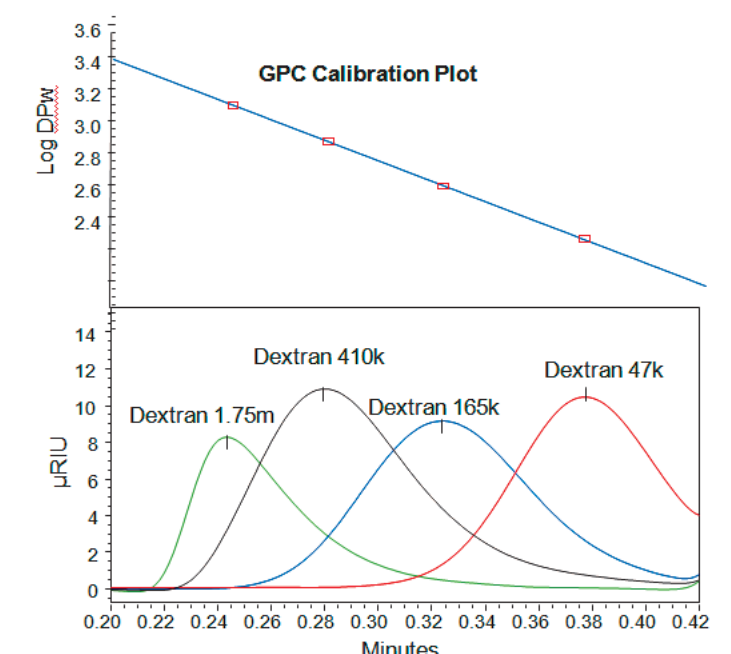
Separation of PEG



High Resolution Characterization of a Liquid Epoxy Resin



30 Second High Throughput APC Method



Mobile Phase: Neat DMSO (HPLC Grade); Column: 1 x 30mm 450A XT
Flow Rate: 0.5 mL/min; Column Temp: 70C; Injection Volume: 10µL;
RI Temp: 55C; Sample Sol'n's: ~1mg Dextran/mL DMSO (w/ 10%LiCl)

CONCLUSIONS

- APC system with BEH particle column technology enables fast and high resolution GPC separation, which allows rapid method development to obtain reproducible polymer molecular weight characterization results.